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Communication

Isomer-selective complexation of copper(I) ionic salts towards 1- and 2-allylbenzotriazoles. Synthesis and characterization of CuBF₄·2-all-bta·H₂O, CuClO₄·2-all-bta, CuClO₄·1-all-bta·2-all-bta and CuHSO₄·2-all-bta – The first known example of CuHSO₄ π -complexes

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ABSTRACT

The alternating current electrochemical synthesis starting from ethanol solution of $Cu(ClO_4)_2 \cdot 6H_2O$, $CuSO_4 \cdot 5H_2O$ and $Cu(BF_4)_2 \cdot 6H_2O$ with the equimolar mixture of 1- and 2-allylbenzotriazole (all-bta) has led to a formation of Cu[2-all-bta]ClO₄ (I), Cu[2-all-bta]HSO₄ (II) and Cu[2-all-bta]BF₄·H₂O (III) compounds. The direct interaction between $Cu(ClO_4)_2 \cdot 6H_2O$ and the mixture of 1- and 2-allylbenzotriazole in ethanol solution results in an appearance of Cu[(1-all-bta)(2-all-bta)]ClO4 (IV) compound. These results are strikingly different from earlier performed syntheses using the same ligands mixture and copper(II) halides, and producing coordination compounds with 1-allylbenzotriazole only. Compounds I and II are isotypical and crystallize in a monoclinic space group Cc. I: a = 9.5413(10) Å, b = 12.3171(9) Å, c = 10.3264(10) Å, $\beta = 111.155(4)^{\circ}$, V = 1131.78(18) Å³, Z = 4. II: a = 9.1707(17) Å, b = 13.6639(17) Å, c = 9.4543(17) Å, $\beta = 105.555(7)^{\circ}$, V = 1141.3(3) Å³, Z = 4. The main feature of structures I and II is a chelatebridging role of the ligand moiety, bonded to one copper ion via C=C-bond of the allyl group and nitrogen atom of the triazole core, and to the second Cu⁺ centre by another N atom. Trigonal-pyramidal copper environment comprises of two nitrogen atoms from different ligand units, C=C-bond and oxygen atom at the apical position. The bridging function of both Cu⁺ cations and 2-all-bta molecules results in the formation of infinite chains. High affinity of BF_4^- anion to the H_2O leads to a formation of compound III including water molecule. It crystallizes in an orthorhombic Pbca space group, a = 13.502(8) Å b = 11.299(5) Å c = 16.124(8) Å, V = 2460(2) Å³, Z = 8. The ligand moiety plays the same as in I and II chelatebridging function, but Cu⁺, being also bonded to C=C group and to two N atoms, is connected with the disordered BF₄ anion through the water bridge. In the crystal structure IV the metal ion possesses mixedisomer surrounding, being bound to N-atom and C=C-bond of 2-all-bta molecule, N-atom of 1-all-btamoiety and $O(ClO_4^-)$ atom at apical position. IR spectra confirm rather effective Cu-(C=C) bonding. © 2012 Elsevier B.V. All rights reserved.

1. Introduction

 Cu^+ is a well-known cation capable to η_2 -coordination with a double carbon-carbon bond. Due to instability of copper-olefin adducts, substituted derivatives of unsaturated hydrocarbons appear to be more suitable ligands for obtaining and investigations of copper(I) – π -adducts. *N*-allylbenzotriazole attracts an attention as a potential ligand with various coordination abilities. Earlier some complexes of copper-halides derivatives of 1-allylbenzotriazole have been obtained and X-ray structurally investigated [1–3] using a mixture of 1- and 2-N-allylbenzotriazole isomers as a starting material. Unexpectedly, using the same mixture of 1- and 2-N-allylbenzotriazoles, complexes of copper(I) ionic salts containing predominantly 2-isomer have been synthesized. The results are represented in this paper.

2. Material and methods

2.1. Synthesis

2.1.1. Synthesis of N-allylbenzotriazole

The ligand was synthesized (according to a procedure similar to that described in [4]) from the equimolar amounts of benzotriazole

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(Reachim), freshly distilled allyl chloride, and NaHCO₃ in ethanol (Fig. 1). The mixture was boiled for 11 days (with breaks for nights) with a reflux condenser and then filtered. A NMR spectrum of used ligand, agreed well with earlier described one, has shown the presence of both 1- and 2-isomers of *N*-allylbenzotriazole in approximately equimolar ratio. A concentrated solution of the mixture of 1-allylbenzotriazole and 2-allylbenzotriazole was used for syntheses of compounds **I**–**IV**.

2.1.2. Synthesis of I-III compounds

High-quality single crystals of all three compounds were obtained by alternating current electrochemical synthesis [5] in ethanol. Solutions contained Cu(ClO₄)₂·6H₂O, CuSO₄·5H₂O and Cu(BF₄)₂·6H₂O respectively (1.0 mmol) and *N*-allylbenzotriazole (2.0 mmol) were placed in small test-tubes. A copper wire was wrapped into a spiral of ~1 cm diameter. A straight copper wire was placed inside the spiral. These copper electrodes were inserted in cork and immersed in the above mentioned solutions. Syntheses were performed using alternating current of 50 Hz, voltage 0.4 V was applied both wire electrodes. After two weeks, colourless crystals of compounds appeared on the electrodes.

2.1.3. Synthesis of compound IV

The reaction conditions were identical to those in the case of **I**, but current was not applied. Colourless crystals have grown on the copper wire after one month. Crystals appear to be enough stable in dry atmosphere. The density of the crystals measured by the flotation method in a mixture of chloroform–bromoform was 1.88, 1.85, 1.77 and 1.61 g/cm³ for **I–IV** respectively.

2.2. Crystallography

Single-crystal data were collected on a Rigaku AFC7 diffractometer equipped by Mercury CCD area detector using graphite monochromatized MoK α radiation. Data treatments were performed using Rigaku CrystalClear software suite program package [6]. All structures were solved by direct methods using SIR-92 [7] program (teXan crystallographic software package of Molecular Structure Corporation [8]) and refined with SHELXL-97 [9] software, implemented in program package WinGX [10]. Hydrogen atoms of ligand moieties were included on idealized positions and refined with geometrical restrictions, H-atoms of water molecule in III and HSO $_{\overline{4}}$ anion in II were found on the difference Fourier maps. Figures were prepared using DIAMOND 3.1 software [11].

2.3. IR spectroscopy

FTIR spectra of **L** and complexes **I**–**IV** were recorded with using of Perkin Elmer GX spectrometer. The resolution in each experiment was 2 cm⁻¹. 1–2 mg of each complex was mixed and grinded with 100 mg of potassium bromide (spectroscopic purity)



Fig. 1. Scheme of the ligand synthesis.

and further the mixtures were pressed into the pellets which had nearly 1 mm of width. Prepared pellets were used to record the spectra of the above mentioned complexes. To record the spectrum of liquid ligand **L** 0.1 mm potassium bromide cell has been used.

2.3.1. Ligand *L* (mixture of 1- and 2-allyl derivatives)

IR: $\nu = 3088(m), 3067(m), 3022(m), 2988(m), 2933(m), 1645(m), 1615(m), 1591(w), 1566(m), 1496(m), 1454(m), 1435(w), 1422(m), 1327(m), 1277(m), 1266(m), 1230(m), 1192(w), 1160(m), 1093(w), 990(m), 935(s), 850(m), 783(m), 746(s), 432(w) cm^{-1}$.

2.3.2. Compounds I-IV

I: IR (KBr): $\nu = 3494(s)$, 3101(w), 3037(w), 2975(m), 2926(m), 2844(m), 2723(w), 1895(w), 1724(m), 1635(s), 1591(m), 1557(w), 1499(m), 1449(s), 1413(w), 1376(w), 1360(w), 1297(w), 1271(w), 1239(m), 1085(s), 998(w), 947(w), 841(m), 786(m), 768(s), 665(w), 577(w), 534(w), 522(m), 434(m) cm⁻¹.

II: IR (KBr): $\nu = 3092(w)$, 2975(m), 2929(m), 2872(m), 2842(m), 2723(w), 1725(s), 1645(w), 1572(m), 1444(s), 1377(m), 1329(m), 1167(s), 1108(m), 1061(m), 998(m), 973(m), 949(m), 841(m), 747(s), 620(s), 592(m) cm^{-1}.

III: IR (KBr): $\nu = 3090$ (m), 3064 (m), 3031 (m), 2983 (m), 1645 (m), 1614 (m), 1607 (m), 1594 (m), 1572 (s), 1496 (m), 1456 (s), 1444 (m), 1422 (m), 1328 (m), 1318 (m), 1306 (m), 1283 (s), 1230 (s), 1200 (w), 1088 (s), 948 (m), 854 (m), 787 (m), 747 (s), 534 (s), 522 (s).

IV: IR (KBr): $\nu = 3170$ (w), 3100 (w), 3035 (w), 2984 (w), 2954 (w), 2924 (m), 2871 (w), 2854 (m), 1644 (m), 1617 (w), 1611 (w), 1594 (m), 1579 (m), 1572 (m), 1495 (m), 1458 (m), 1444 (m), 1423 (m), 1296 (m), 1282 (m), 1230 (m), 1072 (s), 952 (m), 855 (m), 788 (m), 747 (s), 678 (m), 533 (s), 522 (s).

2.4. NMR measurements

¹H NMR spectra (300 MHz) for **I–III** compounds dissolved in DMSO- d_6 were recorded using an Inova 300 Varian spectrometer. Unfortunately, all three compounds are low-soluble in "soft" solvents, including THF- d_8 and acetone- d_6 . The signals from –CH= CH₂ protons were found at the same positions as in free ligand, indicating unquestionably breaking the Cu^I–(C=C) bond because of strong solvating abilities of used solvent. The data were not further analysed, and there is no more discussion about these results.

3. Results and discussion

3.1. Crystal structure

In structures I and II 2-allylbenzotriazole fully realizes its coordination abilities, being bound to one of metal atoms through the N1 atom of triazole core and by C=C-bond of allyl group, and to another metal centre through the N3 atom (Fig. 1). Therefore, the ligand moiety plays in these structures chelating – bridging role. Copper environment comprises two nitrogen atoms from two ligand moieties, the C=C group and the oxygen atom from the anion. In both I and II compounds the metal coordination sphere demonstrates strong tetrahedral motiff, more common for Cu⁺ σ complexes rather than for π -derivatives. In I the Cu–O (ClO₄) distance is slightly elongated in comparison with both Cu-N bond lengths, in **II** the Cu–O (HSO₄) bond length appears to be practically equal to both Cu-N distances (Fig. 2, Tables 1 and 2). The Cu atom is removed from the N1, N3, m (m is a mid-point of C8=C9 bond) plane for 0.38 Å in I and 0.54 Å in II, and the C=C-bond is tilted from the basal plane of coordination polyhedron on 14° in I and 11° in II respectively. A small elongation of coordinated C=Cbonds and tetrahedral rather than trigonal-pyramidal shape of the

Table 2

Cu1-N3

Cu1-N1^a

I



Fig. 2. Metal coordination and ligand surrounding in crystal structure II (structure I differs only by the presence of ClO_{4}^{-} anion instead of HSO_{4}^{-} one).

coordination surrounding indicate a weakness of the Cu-(C=C) interaction. Such a conclusion has an additional confirmation in a rather small (24 $\rm cm^{-1}$ and 42 $\rm cm^{-1}$ in I and II respectively) shift of the C=C stretching frequency. This frequency decreases up to 100 cm^{-1} in copper(I) complexes with ethylen [12]. The steric hindrances lead to a marked difference in values of O4-Cu1-N3 and O4-Cu1-N1 angles (Table 2). Due to the bridging function of both Cu⁺ cations and 2-all-bta molecules infinite V-shaped chains oriented along [101] direction appear. Each two benzotriazole skeletons from two ligand moieties attached to the same copper atom are tilted on 57°. These chains, in turn, are associated into 3D structure by non-valent interactions. Perchlorate-anions, being attached to the chains via the only one oxygen atom demonstrate a noticeable libration, resultated in the enlarged thermal parameters of other three O atoms.

In III the ligand unit plays the same chelating-bridged role (Fig. 3) as in previously described compounds I and II. But rather common tendency of BF_4^- units to be bound with the $3d^{10}$ metal cations through the water bridges [13,14] involves oxygen atom of the water molecule into copper coordination sphere. Despite

Cui-Co	2.095(6)	NI –CuI–III	99.Z(Z)
Cu1-C9 ^a	2.172(6)	N3-Cu1-O4	95.9(2)
Cu1-04	2.234(5)	2.234(5) N1 ^a -Cu1-O4	
Cu1-m	2.025(6)	m-Cu1-04	108.6(2)
Cl1-02	1.404(6)	C8 ^a -Cu1-C9 ^a	36.8 (3)
Cl1-03	1.422(6)		
Cl1-01	1.426(6)		
Cl1-04	1.437(5)		
II			
Cu1-N3	1.973(5)	N3-Cu1-N1 ^a	120.8(2)
Cu1-N1 ^a	2.072(5)	N3-Cu1-O4	101.1(2)
Cu1-04	2.111(4)	N1 ^a -Cu1-O4	104.3(2)
Cu1–C8 ^a	2.135(7)	m-Cu1-O4	111.3(1)
Cu1-C9 ^a	2.166(7)	N1 ^a -Cu1-m	98.7(2)
Cu1-m	2.040(8)	N3–Cu1–m	119.9(2)
S1-03	1.426(6)	C8 ^a -Cu1-C9 ^a	36.9(3)
S1-02	1.436(5)		
S1-04	1.448(5)		
S1-01	1.579(5)		
01-H1	0.95		
III			
Cu1-N1	1.976(3)	N1-Cu1-N3 ^b	118.24(14)
Cu1–N3 ^b	2.055(3)	N1–Cu1–m	129.0(1)
Cu1–C8 ^b	2.095 (4)	N3 ^b -Cu1-m	98.59(9)
Cu1–C9 ^b	2.141(4)	N1-Cu1-O1	97.8(1)
Cu1-01	2.166(3)	N3 ^b -Cu1-O1	96.7(1)
Cu-m	2.007(4)	m-Cu1-O1	112.32(8)
		C8 ^b –Cu1–C9 ^b	37.3(2)
IV			
Cu1-N11	1.947(3)	N11-Cu1-N21	112.47(14)
Cu1-N21	2.020(3)	N11–Cu1–m	141.3(1)
Cu1-C28	2.058(4)	N21–Cu1–m	100.17(8)
Cu1-C29	2.094(4)	N11-Cu1-O4	92.3(1)
Cu1-04	2.452(4)	N21-Cu1-O4	101.0(1)
Cu1-m	1.962(7)	m-Cu1-04	101.6(8)
Cl1-02	1.383(4)	C28-Cu1-C29	38.1(2)
Cl1-03	1.386(5)		
Cl1-01	1.429(3)		
Cl1-04	1.442(3)		

Selected bond distances (Å) and angles (°) in structures I–IV.

1.959(5)

2.034(6)

N3-Cu1-N1^a

N3-Cu1-m

Symmetry codes: (a) x + 1/2, -y + 1/2, z + 1/2.

^b Symmetry codes: (b) -x + 3/2, y - 1/2, z.

Table 1

Details of experimental and crystallographic data for compounds I-IV.

Compound	I	П	III	IV
Formula	C9H9ClCuN3O4	C9H10CuN3O4S	C9H11BCuF4N3O	C18H18ClCuN6O4
Fw	322.18	319.8	327.56	481.38
T (K)	200	293	200	200
Crystal system	Monoclinic	Monoclinic	Orthorhombic	Triclinic
Space group	Сс	Сс	Pbca	ΡĪ
a (Å)	9.5413(10)	9.1707(17)	13.502(8)	7.8621(6)
b (Å)	12.3171(9)	13.6639(17)	11.299(5)	10.2633(7)
c (Å)	10.3264(10)	9.4543(17)	16.124(8)	13.1327(10)
α (deg)				97.485(2)
β (deg)	111.155(4)	105.555(7)		106.141(3)
γ (deg)				99.838(2)
$V(Å^3)$	1131.78(18)	1141.3(3)	2460(2)	985.10(13)
Ζ	4	4	8	2
λ (Å)	0.71069	0.71069	0.71069	0.71069
μ (mm ⁻¹)	2.176	2.107	1.819	1.284
D _{calcd} (g/cm ³)	1.891	1.861	1.769	1.623
$R_1^{\mathbf{b}}$	0.0511	0.0455	0.0682	0.0694
wR ₂ ^c	0.1067	0.1147	0.2048	0.2104
GOF ^a	1.088	1.133	1.208	1.147

^a $R_1 = \sum ||F_0| - |F_c| \sum / ||F_0|$ ^b $wR_2 = [\sum w(F_0^2 - F_c^2)^2 / \sum (w(F_0^2)^2)]^{1/2}$. ^c $GOF = [\sum w(F_0^2 - F_c^2)^2 / (N_0 - N_p)]^{1/2}$, where N_0 = no. of refins and N_p = no. of refined parameters.



Fig. 3. Metal coordination and ligand surrounding in crystal structure III.

121.7(2)

127.9(2)

a neutral state this oxygen atom forms rather short (2.166(3) Å) Cu–O bond. This value is close to the earlier observed one of 2.145(7) Å in the structure of CuBF4·2C₆H₄N₃(OC₃H₅)·H₂O [13]. The metal coordination sphere is very similar to those in I and II and also demonstrates strong tetrahedral distortion with copper atom removed from the basal plane by 0.44 Å. The coordinated C=Cbond is moderately elongated to 1.353(6) Å. The C=C stretching frequency is shifted on 42 cm⁻¹, similarly to that in I and II. The BF₄ anion demonstrates rather common orientantional disorder. Infinite (CuBF₄·2-all-bta)_n chains are interconnected by O–H···F hydrogen bonds into layers lying in (110) plane.

Contrary to the above compounds discussed with 2-all-btz as a ligand the complex IV represents an unusual case of mixedisomer coordination compound. In contrast to compounds I-III, the 2-allylbenzotriazole molecule acts in the structure of **IV** as a chelate ligand, being bonded to the copper atom through the nitrogen atom and a C=C-bond of allyl group. Two other places in the metal surrounding are occupied by nitrogen atom from the molecule of 1-allylbenzotriazole and by moderately removed oxygen atom belonging to perchlorate-anion (Fig. 4, Table 2). The Cu–O(ClO₄) distance of 2.425(5) Å noticeably differs from that in compound I. This value, as same as earlier observed $Cu-O(ClO_4)$ distance of 2.69(1) Å in Cu(C₇H₁₀N₂)ClO₄ [15] agrees much better than that in I with the hardness, using Pearson terms, of ClO_4^- anion and softness of Cu⁺ cation [16]. Two benzotriazole cores belong to 1- and 2-allylbenzotriazole units, attached to the same copper centre, are mutually tilted on 22.9(1)°. Because 1-all-bta moiety is bound with the metal centre via N atom only. uncoordinated allyl group undergoes small disordering, which is seen in an apparent shortening of C28=C29 bond to 1.25 Å. Because of the presence in this structure both coordinated and uncoordinated allyl groups the IR spectrum of **IV** contains peaks at 1617 cm^{-1} (uncoordinated) and 1572 cm⁻¹ C=C stretching frequencies. Isolated [Cu(1-all-bta)(2all-bta)ClO₄ units are interconnected by weak interactions only.

3.2. Isomer-selective complexation

As it is known, the simplest technique to obtain an N-allylbenzotriazole – a reaction between benzotriazole and allyl halides, resulting in a formation of a mixture of 1- and 2-allyl



Fig. 4. Metal coordination and ligand surrounding in crystal structure IV.

derivatives [17]. An NMR experiment has confirmed a presence of both isomers in the product. This mixture was used for further synthesis of coordination compounds of copper(I) chloride, bromide, nitrate, tetrafluoroborate, perchlorate and hydrosulfate. Under very similar reaction conditions the only derivatives of 1all-bta with CuCl, Cu(Cl, Br), and CuNO₃ were obtained. Contrary, electrochemical synthesis using copper(II) perchlorate, tetrafluoroborate and sulphate resulting in a formation of complexes with 2-allylbenzotriazole. One may suggest that the main reason of such selectivity is an entropy factor. In the case of weakly bonded anions $-ClO_{4}$, BF₄ and HSO₄, because of high accessibility of the metal centre 2-allylbenzotriazole fully realizes it coordination abilities and plays chelate-bridging role. It is well known, that chelate-effect strongly increases complex stability. Such chelatebridging coordination mode requires three places (from four available) in the copper environment. On the other hand, halideanions in the structure play usually bridging role, occupy at least two places in the metal coordination sphere and, therefore, retains only two places in metal surrounding, preventing energetically favourable chelate-bridging ligand coordination. The nitrate-anion frequently behaves in the same manner. Presence of two accessible places in the Cu⁺ coordination sphere promotes coordination of either one ligand moiety in chelate mode or two ligands in bridging manner, in the last case an adduct should be less soluble and, consequently, easier crystallized. One may also note, that the 1-allylbenzotriazole is more suitable ligand for the bridging coordination mode (which requires usually one or two places in copper environment) because of higher spatial accessibility of the N1 atom of triazole core comparatively with that for 2-isomer. That is the reason why only 1-all-bta derivatives appear with copper(I) halides and nitrate.

The formation of two different (I and IV) compounds from the same starting materials attracts an additional attention. The standard electrode potentials for the Cu^{2+}/Cu^+ pair is 0.16 V (in aqueous media). Because of 1) specific coordination ability of Cu⁺ towards carbon C=C double bond, and 2) sharp increase of formation constants in the case of N, C=C – chelate metal coordination one may suppose, that this electrode potential could easily be shifted into negative area, i.e. reaction may run without applying additional voltage. But such a reaction should be much slower than that under applied tension. It means that Cu⁺ concentration in solution is much lower than the concentration of the ligand, resulting in a high probability of a formation of single (solvated and/or coordinated by ligand) metal ions. Coordination under such conditions should produce the most thermodynamically stable moieties, i.e. bonded in a N, C=C-chelate manner metal centres. The presence of anion in copper coordination sphere is a requirement of the charge balance. The molecule of 1-allylbenzotriazole could compete more successfully for the fourth place in the metal environment because of higher spatial accessibility of the N3 atom comparatively with the 2-derivative. Finally, one may expect a formation of isolated complex units, as was in reality observed. The reaction under applied tension should run much quicker and generate higher amount of Cu⁺ ions. These conditions are favourable for a coordination of ligand moiety with more than one metal ion and, consequently, for the appearing of polymeric structures. There are two reasons why under such conditions only crystals of 2allylbenzotriazole complex appear. At first, mentioned above thermodynamic stability of the complex with chelate metal coordination. Additionally, bridging function of 1-allylbenzotriazole in hypothetical (Cu - 1-allylbenzotriazole)_n chains requires only two places in the copper coordination sphere. All these points increase the probability of appearance during electrochemical synthesis Cu(I)-complexes of 2-allylbenzotriazole, as was also observed experimentally.

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Appendix A. Supplementary material

CCDC 853613 (I), 853615 (II), 853612 (III), 853614 (IV) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/ conts/ retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033; e-mail: deposit@ccdc. cam.ac.uk).

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